Journal of Pure and Applied Sciences (JPAS),
Volume 2, Issue 2, Page 124-135, 2026

Groundnut Shell-Derived Amorphous Silica as a Sustainable Alternative to Quartz in Porcelain Stoneware: Phase and Microstructural Evaluation

E. T. Aliu[1*], S. S. Owoeye[2], Y. D. Lamidi[3]

[1,2,3]Department of Glass and Ceramic Technology, The Federal Polytechnic Ado-Ekiti, Ekiti State, Nigeria.
[1]aliu_et@fedpolyado.edu.ng, [2]owoeye_ss@fedpolyado.edu.ng, [3]lamidi_yd@fedpolyado.edu.ng
*Corresponding author (Email: owoeye_ss@fedpolyado.edu.ng, +2348064461915)

ABSTRACT

This study investigates the development of eco-friendly porcelain stoneware bodies using amorphous silica derived from groundnut shell ash (GSA) as a sustainable alternative to conventional quartz. Groundnut shells were subjected to controlled combustion and subsequent chemical precipitation to synthesize high-purity amorphous silica (GSASi). The extracted silica exhibited a high silica content of 93.7 wt.% and an amorphous structure confirmed by X-ray diffraction (XRD). Two stoneware compositions were formulated: one containing commercial quartz (PSWTSS) and the other incorporating GSASi (PSWTAS) as a complete replacement for quartz. The samples were prepared via conventional ceramic processing and sintered at 1050°C and 1100°C. Phase analysis revealed the presence of quartz, mullite, and albite in both compositions, indicating that the substitution did not adversely affect the essential phase formation. Microstructural evaluation using scanning electron microscopy (SEM) showed that the GSASi-based sample exhibited a finer, more homogeneous, and less porous structure compared to the quartz-based counterpart, especially at 1100°C. This improvement is attributed to the higher reactivity of amorphous silica, which enhances densification and promotes mullite formation. The findings demonstrate that groundnut shell-derived silica is a viable and sustainable substitute for conventional silica in porcelain stoneware production, with potential benefits in improving microstructural properties and reducing environmental impact.

***Keywords*— groundnut shell ash, amorphous silica, chemical precipitation, sintering, mullite, stoneware, XRD, SEM.**

I. INTRODUCTION

The growing demand for sustainable materials in the ceramic industry has encouraged the exploration of alternative raw materials from renewable sources. One promising approach is the utilization of agro-waste silica in the production of stoneware bodies. Stonewares are dense, vitrified ceramics characterized by low water absorption, high mechanical strength, and excellent chemical resistance, making them ideal for both wall and floor applications [1]. Traditionally, their composition includes kaolin, ball clay, feldspar, and quartz, which serve as plastic, fluxing, and refractory components, respectively. However, the extraction and processing of these natural raw materials are energy-intensive and environmentally unsustainable [2]. To address these challenges, the use of agro-waste-derived silica offers a viable, eco-friendly alternative that can significantly reduce production costs and environmental pollution while maintaining or even enhancing the desired properties of the stoneware bodies [3].

Agro-wastes such as rice husk, sugarcane bagasse, bamboo leaves, and palm kernel shells are rich in silica and are generated in large quantities in agricultural regions [4]. When properly processed, these wastes can be converted into high-purity silica ash suitable for ceramic production. Rice husk ash (RHA) is the most widely studied agro-waste source because it contains up to 90–95% amorphous silica after controlled combustion [5]. Similarly, sugarcane bagasse ash (SCBA), bamboo leaf ash (BLA), and palm ash also contain considerable amounts of silica with minor impurities like potassium, calcium, and iron oxides [6]. The presence of amorphous silica in these ashes makes them highly reactive, enhancing sintering and densification during the firing of ceramic bodies [7]. Thus, substituting natural quartz with agro-waste silica can improve the vitrification process, promote the formation of glassy phases, and reduce the firing temperature required to achieve optimal mechanical properties.

The typical stoneware composition can be modified to incorporate agro-waste silica by partially replacing the quartz content. For instance, replacement levels of 5%, 10%, 15%, and 20% by weight have been studied in several works [2]. The remaining constituents—kaolin, ball clay, and feldspar—are kept constant to maintain the desired balance between plasticity, fluxing behavior, and refractoriness. The blended powders are homogenized through wet ball milling, dried, and pressed into desired shapes using uniaxial pressing. The green compacts are then fired at temperatures ranging from 1150°C to 1250°C. The firing schedule is optimized to promote densification and vitrification while preventing excessive glass formation or bloating, which can result from excess alkali oxides in the agro-silica [7].

Several studies have shown that substituting a portion of quartz with agro-waste silica can lead to significant improvements in the microstructure and performance of stoneware bodies. The fine amorphous silica particles enhance the reactivity of the ceramic body, leading to improved densification and reduced porosity. Consequently, water absorption decreases, and mechanical properties such as flexural strength, hardness, and abrasion resistance improve. In some cases, the introduction of agro-waste silica has been observed to lower the required firing temperature, thereby saving energy and production costs [6]. However, at higher replacement levels (above 20%), impurities such as alkali and alkaline earth oxides may induce excessive liquid phase formation, which can cause deformation, bloating, or reduced mechanical strength. Therefore, an optimal replacement level must be established through experimental evaluation to balance performance and processing stability.

From an environmental and economic standpoint, the utilization of agro-waste silica in stoneware production presents several benefits. It reduces the volume of agricultural waste that would otherwise contribute to environmental pollution through open burning or landfilling [3]. The approach also decreases dependency on mined quartz, conserving natural resources and promoting a circular economy. Additionally, since agro-wastes are often locally available in large quantities, their use can reduce raw material transportation costs, contributing to a lower carbon footprint [7]. This approach aligns with sustainable development goals by promoting waste valorization, environmental protection, and resource efficiency.

Due to these numerous advantages offered by agro-wastes silica, concerted efforts have been tailored towards exploring other agro-wastes with promising potential for silica extraction and assess its inclusion in stoneware body. To the best of our knowledge, groundnut shell-derived amorphous silica has not previously used as a complete replacement for quartz in porcelain stoneware. Hence, the present work aims to replace quartz completely in porcelain stoneware using amorphous silica derived from groundnut shell (GSASi). The morphological and phase analysis of the developed stoneware bodies were then analyzed.

II. MATERIALS AND METHODS

A. Processing of raw materials

All raw materials used in this research were sourced locally to promote cost-effectiveness and sustainability. Kaolin, feldspar, clay and quartz were all obtained locally, while the groundnut shell used was gotten from the Federal Polytechnic Ado-Ekiti. The obtained raw materials were adequately beneficiated to obtain high purity materials before use.

B. Extraction of amorphous silica from groundnut shell ash (GSA)

Extraction of amorphous silica from groundnut shell ash (GSA) is a well-established process in green chemistry and materials science. Groundnut (peanut) shells are rich in silica (SiO_2), which can be recovered in amorphous form through controlled burning and chemical treatment.

After the groundnut shells were air-dried, the groundnut shells were burnt by means of open burning. Then, it was fired inside kiln at the temperature of 800oC for proper combustion result. Groundnut shells ash was obtained after this process.

Sodium hydroxide of 30g poured in 250 ml of distilled water to obtain 3 Mol of NaOH, the result solution was reacted with 15g at groundnut shell ash in Erlenmeyer flask at 200oC for 2 hours with continuous stirring with the aid of magnetic stirrer bar. After heating, was filtered out using filter paper, result obtained is called sodium silicate solution. 3M Hydrochloric acid was reacted with sodium silicate solution to precipitate the bio silica while stirring continuous until white content begins to form. It was then allowed to age for 18 hours before filtering. The filter silica gel was then washed with distilled water several times to get clearer and purer silica. The silica particles were then oven dried at the temperature 80oC for 12 hours.

C. Samples Preparation

In this study, standard porcelain stoneware body comprises of 50 wt. % clay, 40 wt. % feldspar and 10 wt. % quartz was used as the standard composition. The materials used were all received in their already processed powder form and do not require further processing. The ball clay ensures plasticity while quartz is used to strength to the body. Feldspar is used as flux, to aid sintering by introducing glassy phase that ensure solid sintering. Initially, each material was accurately weighed using digital weighing balance. The accurately weighed materials were then blended together thoroughly in the porcelain-lined mortar with the aid of pestle. Afterward, further mixing was carried out using porcelain-lined ball mill with addition of ethanol (250 ml) to ensure thorough blending of the materials together.

The use of ethanol is preferable to water in order to ensure purity and to hasten the drying of the resultant slurry material after ball milling. The slurry was later dried in an oven at 110oC until dried body is obtained. The thoroughly blended materials are then compressed inside a steel mould to form compact disc-like samples (20 x 10 mm) under a force of 40 kN. The pressed samples were then allowed to dry under ambient (room) temperature, followed by drying inside an electric oven at temperature of 110oC. The pressed samples were then arranged inside a microwave kiln box and placed in a microwave oven (Model P90N30EP-ZK) operating at a frequency of 2.45 GHz and a power output of 900 W for sintering. The sintering of the samples was achieved at temperatures of 1050 and 1100oC, respectively. After sintering, the samples were then allowed to cool inside the kiln box to room temperature before offloading. Table 1 shows the composition of the porcelain in weight percentage.

Journal of Pure and Applied Sciences (JPAS),
Volume 2, Issue 2, Page 124-135, 2026

D. Characterization

Complete solar-cell devices were fabricated in a superstrate configuration: glass/ITO (~15 Ω/□)/ZnO/ZnS/CZTS/Ag. ITO-coated glass (15 Ω/□ sheet resistance, AMTECH) was cleaned by sequential ultrasonic baths in detergent, DI water, acetone, and isopropanol, then dried with N_2. The spray-deposited ZnO (50 nm) and ZnS (100 nm) layers were applied as above. The CZTS absorber (500 nm) was then deposited directly on top of the ZnS. Finally, Ag contact pads (~100 nm thick) were thermally evaporated through a shadow mask to define ~0.1 cm^2 device area. The device structure ensures light enters through the ITO/glass side, passing the ZnO/ZnS window layers before reaching the CZTS absorber.

E. Characterization

The formed samples were only characterized for their phase and microstructure characteristics. The phase constituents were characterized by means of X-ray diffractometry (XRD), while scanning electron microscopy with Energy Dispersive Spectroscopy (SEM-EDS) was used to study the microstructural property.

III. RESULTS AND DISCUSSION

A. Chemical Composition (Extracted Silica)

Table 2 shows the result of the chemical composition of the groundnut shell ash (GSA) and amorphous silica powder precipitated from the GSA (GSASi) respectively. The result showed that the synthesized silica from the groundnut shell ash (GSA) has the high silica content of 93.7 wt. % compared with 79.6 wt. % obtained for GSA. This justifies the chemical precipitation procedure used to produce the synthesized silica. The presence of aluminum oxide (Al2O3) detected is confirmed by XRD with detection of orthoclase. Its presence in the amorphous silica also shows its incomplete removal during chemical precipitation.

Table 1. Composition of the porcelain tile body in weight percent

Samples	Ball clay wt.%	Kaolin wt.%	Feldspar wt.%	Quartz wt.%	Amorphous silica (GSA_{Si}) wt.%
PSWTSS	15	35	40	10	0
PSWTAS	15	35	40	0	10

Journal of Pure and Applied Sciences (JPAS),
Volume 2, Issue 2, Page 124-135, 2026

Table 2. Composition Analysis (oxide composition) in Weight Percent

	SiO_2	CaO	MgO	Fe_2O_3	Al_2O_3	MnO	K_2O	P_2O_5
GSA	79.6	3.34	-	1.53	2.11	0.27	4.05	6.67
GSA_{Si}	93.7	1.56	-	0.15	2.11	0.03	-	-

B. Phase Analysis (Extracted Silica)

Figure 1 shows the result of the superimposed X-ray diffraction analysis for the GSA and GSASi respectively. This result showed that the groundnut shell ash (GSA) exhibited predominant crystalline peak at 27o theta and minor crystalline peak at 50o theta respectively. This predominant crystalline peak is identified as quartz, and which is in agreement with previously reported works on other agro-waste ashes [8]. The other minor peak of Orthoclase result as a natural constituent of most agro-waste materials. However, for the GSASi, it is observed that a broad hump diffractogram with no perceptible portion of crystalline peaks is observed. This broad hump structure indicate that the synthesized silica powder is amorphous. This is also in agreement with previous works [9].

C. Microstructure Examination (Extracted Silica)

Figure 2 depicts the microstructure examination of the GSA and GSASi respectively. The GSA showed somewhat fibrous structure while the GSASi showed a high level of particle agglomeration.

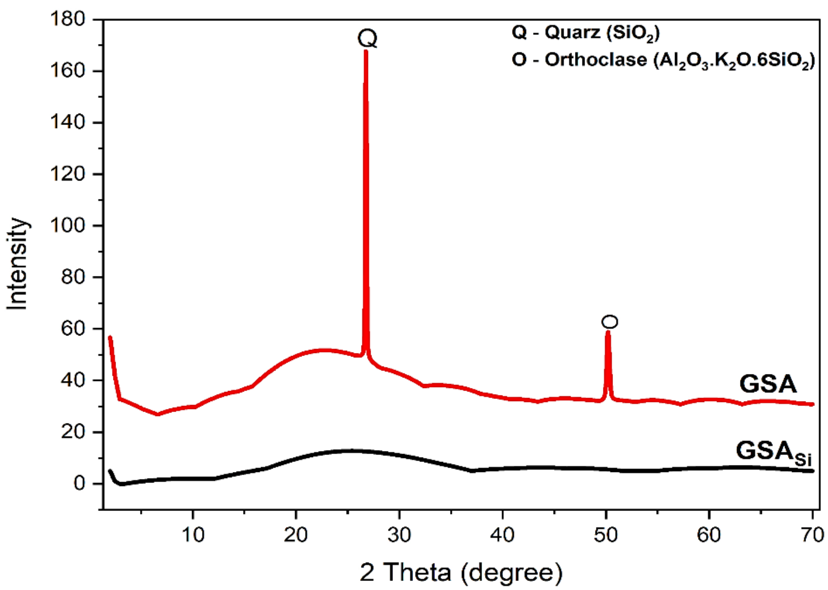
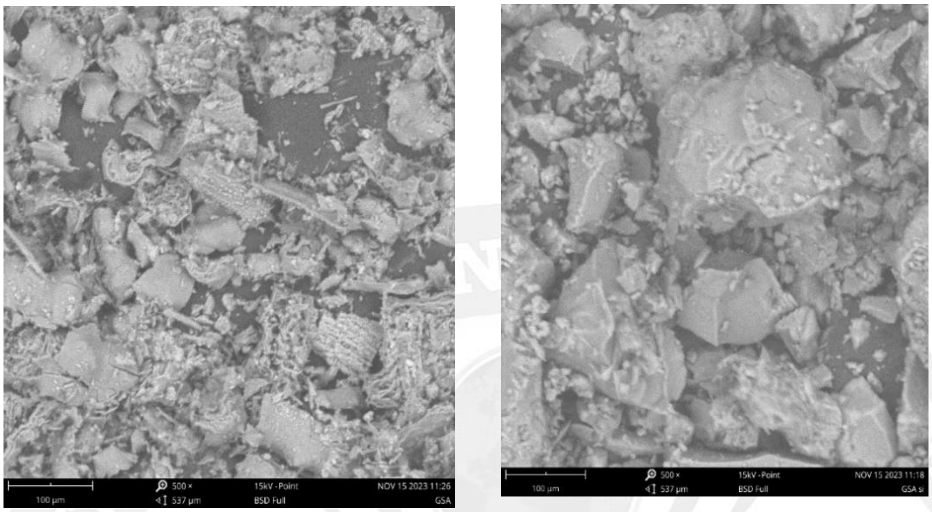


Figure 1: Superimposed XRD of both GSA and GSA_{Si}

Journal of Pure and Applied Sciences (JPAS),
Volume 2, Issue 2, Page 124-135, 2026



(a) **(b)**

Figure 2: Scanning Electron Microscopy Image of (a) GSA (b) GSA$_{S}$i

D. Phase Analysis (Developed Porcelain)

Figure 3 represents the superimposed XRD analysis of the sintered stoneware bodies coded PSWTSS1050, PSWTSS1100, PSWTAS1050 and PSWTAS1100, sintered at 1050 and 1100oC, respectively. All the samples indicate the presence of quartz (SiO2), mullite (3Al2O3.2SiO2), and albite (NaAlSi3O8). A negligible hump is noticed for sample PSWTSS1050 at 2θ = 20 – 35o. The observable hump signifying the presence of glassy phase, as reported by Ochen et al. [10]. Whereas, no noticeable hump is detected for sample containing amorphous silica (GSASi) PSWTAS1050, at sintering temperature of 1050oC. This suggest at sintering temperature of 1050oC, the albite noticeable in the spectra is yet to fully react with predominant quartz to give glassy phase. Furthermore, the no observable hump for sample containing GSASi sintered at 1050oC, might also indicates likelihood of the GSASi not yet reacting in the body to obtain vitreous melt. However, at sintering temperature of 1100oC the broad hump is noticeable for PSWTSS1100, while sample PSWTAS1100 showed broader hump at 2θ = 20 – 35o. This suggests the GSASi showed effective reaction in the stoneware composition at elevated temperature. Thus indicates its promising potential as alternative for commercial quartz in the stoneware body. The noticeable peaks of albite might be due to not much formation of glassy phase probably as a result of sintering temperature adopted in this work. Glassy phase are obtained from reaction of silica with the flux (feldspar) in the stoneware body, and this mostly occurred at temperature ≥1200oC [11-13]. The presence of predominant crystalline phases of both mullite and quartz has been documented in several previous works [11-14]. However, Guzman et al. [15] reported the presence of albite alongside mullite, quartz, and cristobalite.

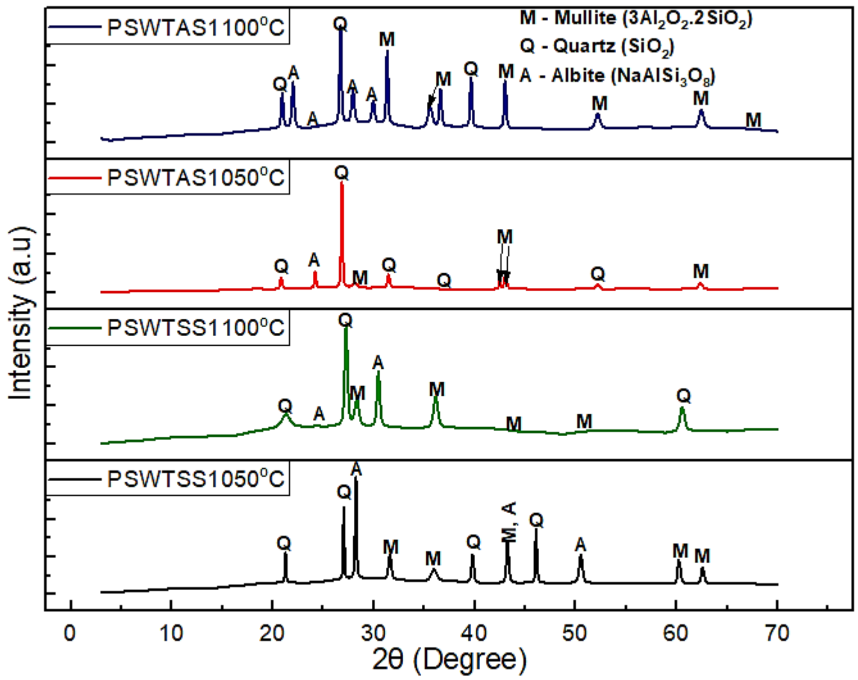


Figure 3: XRD Phase Analysis of the Developed Porcelain Bodies

E. Morphology

Figures 4 (a-c) show the microstructure of the developed stoneware bodies coded PSWTSS1050, PSWTSS1100, and PSWTAS1100, sintered at 1050 and 1100oC, respectively.

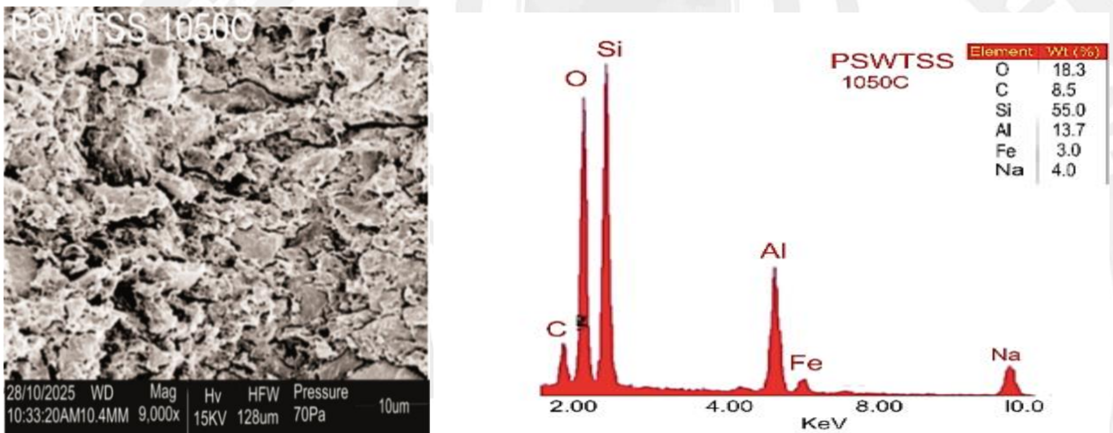
The SEM micrographs of the developed porcelain stoneware bodies reveal the effects of silica source and sintering temperature on microstructural evolution. PSWTSS1050, sintered at 1050°C using conventional quartz, shows a relatively coarse and heterogeneous microstructure with noticeable intergranular porosity. The grains are irregular, and residual quartz particles are clearly seen, suggesting limited mullite formation and incomplete densification at this temperature. However, at elevated sintering temperature of 1100°C, PSWTSS1100 reveals a more close-packed and slightly finer microstructure. The number of residual quartz particles decreases, and early-stage primary mullite formation is evident, indicating improved densification. However, some micro-pores are still noticeable, indicating incomplete vitrification at this intermediate temperature.

PSWTAS1100, in which conventional quartz was replaced with amorphous silica derived from groundnut shells (GSASi), shows a notably finer and more uniform microstructure at the same sintering temperature. Grains are more homogeneously distributed, and intergranular porosity is significantly lowered compared

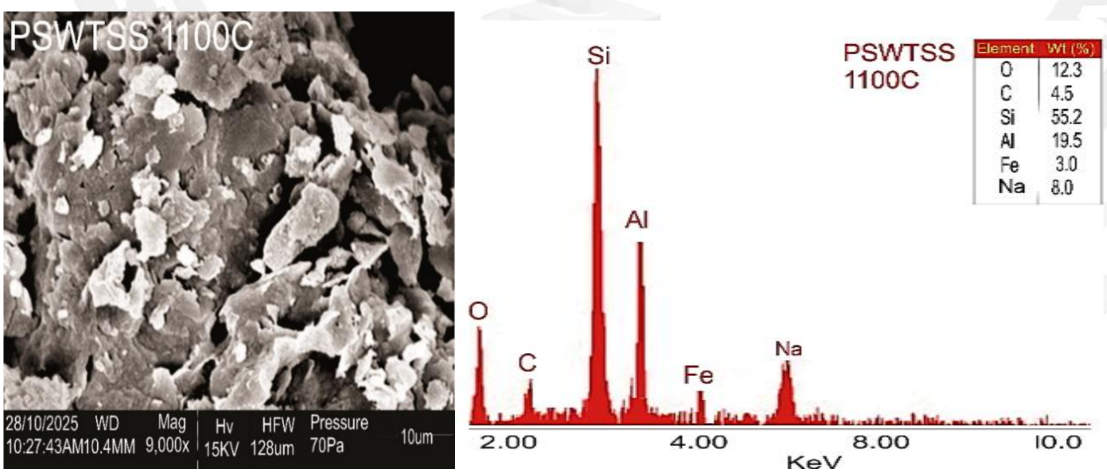
Journal of Pure and Applied Sciences (JPAS),
Volume 2, Issue 2, Page 124-135, 2026

to PSWTSS1100. The absence of unreacted quartz particles suggests that the amorphous silica promotes higher reactivity, facilitating mullite formation and improved densification even at 1100°C.

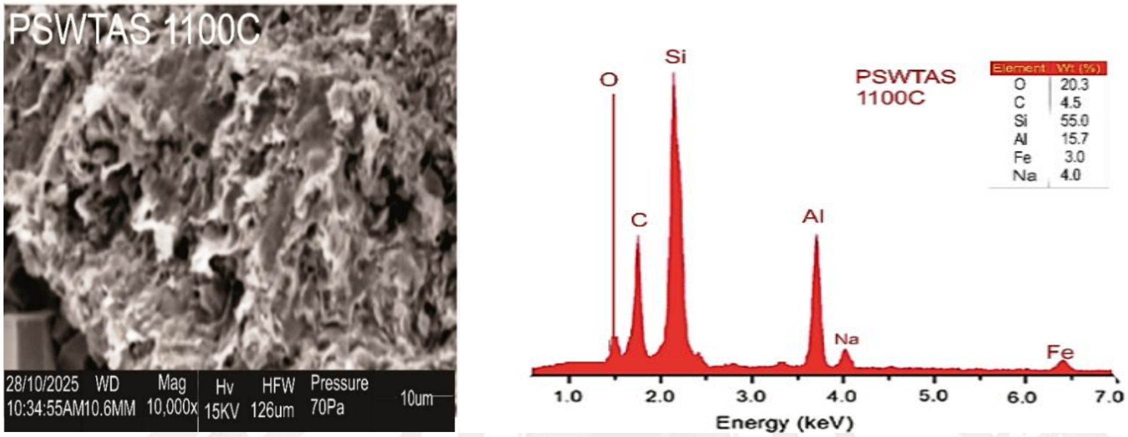
Generally, the SEM analysis demonstrates that both sintering temperature and silica source strongly influence microstructure. Elevated temperature improves densification and mullite formation in traditional quartz-based bodies, while substitution with amorphous agro-waste silica accelerates these processes, producing finer, more homogeneous, and less porous microstructures at equivalent sintering conditions.



(a)



(b)



(c)

Figure 4: SEM Image of sintered stoneware bodies (a) PSWTSS1050 (b) PSWTSS1100 (c) PSWTAS1100

IV. CONCLUSION

In this present work, two stoneware bodies containing traditional quartz and amorphous silica derived from groundnut shells (GSASi) was produced by sintering method at temperatures of 1050 and 1100oC. The morphological and phase characterization was carried out to assess the influence of the amorphous silica in the stoneware body mix. The results showed that:

i. The amorphous silica from the groundnut shells (GSASi) consist of 93.7 wt.% silica, while it exhibited broad hump diffractogram to affirm its amorphous nature.

ii. The phase constituent of both the standard stoneware body (PSWTSS) and the amorphous silica substituted composition (PSWTAS) sintered at 1050 and 1100oC, showed the presence of quartz, mullite, and albite. This suggests the amorphous silica use did not impair the necessary phase relevant to sintered stoneware body, which is in agreement with literature.

iii. The amorphous-silica substituted stoneware body exhibited finer microstructure with considerable mullite formation and vitreous phase, which suggests the high reactivity of amorphous silica compared with conventional silica.

iv. The mullite formation is not well pronounced owing to the sintering temperature range employed.

v. Amorphous silica derived from groundnut shell can find suitable use as silica in development of stoneware body since its use did not impair the necessary phase constituents and morphology.

Acknowledgement

TETFund Nigeria and Federal Polytechnic, Ado-Ekiti.

REFERENCES

[1] S. Chandrasekhar, K. G. Satyanarayana, P. N. Pramada, P. R. Raghavan, and T. N. Gupta, "Review: Processing, properties and applications of reactive silica from rice husk—An overview," Journal of Materials Science, vol. 38, no. 15, pp. 3159–3168, 2003.

[2] M. Dondi, M. Marsigli, and I. Venturi, "Clays and bodies for ceramic tiles: Reappraisal and technological classification," Applied Clay Science, vol. 96, pp. 91–103, 2014.

[3] S. Singh, R. K. Majhi, S. K. Nayak, and B. B. Pradhan, "Utilization of agro-industrial waste for the production of sustainable ceramics," Journal of Cleaner Production, vol. 141, pp. 10–23, 2017.

[4] M. K. Naskar and M. Chatterjee, "A review on the synthesis of silica and its composites from agricultural wastes," Materials and Manufacturing Processes, vol. 33, no. 10, pp. 1031–1050, 2018.

[5] V. P. Della, I. Kühn, and D. Hotza, "Rice husk ash as an alternate source of silica in ceramics: A review," Química Nova, vol. 25, no. 6B, pp. 1116–1121, 2002.

[6] M. Abdullahi, "Characteristics of bagasse ash as a supplementary cementitious material," International Journal of Civil and Structural Engineering, vol. 3, no. 1, pp. 167–175, 2012.

[7] A. Kumar, K. Kumar, N. Kaushik, and S. Sharma, "Renewable energy and sustainable development: A review of the environmental impact of using agricultural waste for various applications," Journal of Environmental Management, vol. 270, Art. no. 110887, 2020.

[8] K. N. Farooque, M. Zaman, E. Halim, S. Islam, M. Hossain, Y. A. Mollah, and A. J. Mahmood, "Characterization and utilization of rice husk ash (RHA) from rice mill of Bangladesh," Bangladesh Journal of Scientific and Industrial Research, vol. 44, no. 2, pp. 157–162, 2009.

[9] S. S. Owoeye, S. M. Abegunde, and B. Oji, "Effects of process variable on synthesis and characterization of amorphous silica nanoparticles using sodium silicate solutions as precursor by sol–gel method," Nano-Structures & Nano-Objects, vol. 25, Art. no. 100625, 2021.

[10] W. Ochen, F. M. D'ujanga, B. Oruru, and P. W. Olupot, "Physical and mechanical properties of porcelain tiles made from raw materials in Uganda," Results in Materials, vol. 11, Art. no. 100195, 2021.

[11] G. A. Álvaro, D. A. Silvio, A. B. Vicente, Q. V. María Francisca, and S. V. Enrique, "Feasibility of using rice straw ash in porcelain tile compositions," in Proc. QUALICER, 2014, pp. 1–11.

[12] S. Kitouni and A. Harabi, "Sintering and mechanical properties of porcelains prepared from Algerian raw materials," Cerâmica, vol. 57, pp. 453–460, 2011.

[13] J. Martín-Márquez, J. Ma. Rincón, and M. Romero, “Effect of firing temperature on sintering of porcelain stoneware tiles,” Ceramics International, vol. 34, pp. 1867–1873, 2008.

[14] J. Martín-Márquez, J. Ma. Rincón, and R. M. Romero, “Mullite development on firing in porcelain stoneware bodies,” Journal of the European Ceramic Society, vol. 30, pp. 1599–1607, 2010.

[15] A. Guzmán, S. Delvasto, E. Sánchez, and V. Amigó, “Cenizas del tamo de arroz como substituto del feldespato en la fabricación de cerámica blanca,” Boletín de la Sociedad Española de Cerámica y Vidrio, vol. 52, pp. 25–30, 2013.